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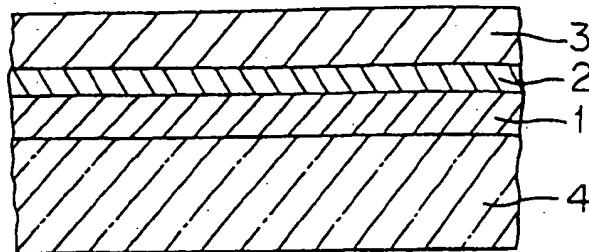
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(54) Method of making a heat treated coated glass.

(57) A method of making a heat treated coated glass comprising the steps of: forming a solar control layer (1) or an electroconductive layer (1) on a glass substrate (4); forming a first protective layer (2) composed of a non-oxide or not completely oxidized, which is transparent in a region of visible light, and which remains transparent even when oxidized, to provide a glass coated with a multi-layer comprising at least two layers including said solar control layer (1) or an electroconductive layer (1) and said first protective layer (2); and performing heat-treatment for said coated glass.

**FIGURE 1**



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This invention relates to a method of making a heat treated coated glass, particularly to a method of making a heat treated coated glass which minimizes changes of characteristics thereof before and after bending thereof.

Conventionally, a bent coated glass is made by performing a coating on a glass which is already bent.

5 Or, it is made by bending a glass coated with a metallic film or a metal nitride film to be protected which is sandwichingly covered with an easily oxidized metal such as tantalum, and in bending thereof, the easily oxidized metal is mainly oxidized, thereby protecting the film to be protected. Or, it is made while preventing the oxidation of the film to be protected by maintaining a nonoxidizing atmosphere during the bending.

10 Among the above technologies, in the method of coating the bent glass, it is not possible to perform a uniform coating on the bent glass, unless the coating process and the coating condition are controlled 15 complying with a configuration of the bent glass. Accordingly, it is difficult to obtain products thereof provided with the high quality and the uniform performance.

15 On the other hand, in the method of sandwiching the layer to be protected by the easily oxidized metal, characteristics of the coated glass change before and after the bending. Therefore, in a plane glass which prepared, or a heat treatment which is the same with that in the bending should be performed. Accordingly, a considerable extravagance is present in the product control.

20 Moreover, in the method of maintaining the nonoxidizing atmosphere during bending, revamping of a normal bending device is necessitated, which increases the production cost. As the heat treating of glass, tempering is generally performed which is similar to bending. With respect to a tempered coated glass, the situation is the same as in the bent coated glass.

25 It is an object of the present invention to provide a method of making a heat treated coated glass wherein a coated plane glass having an excellent uniformity is made by employing a normal coating technology to a plane glass at a low cost, and bending and/or tempering which is generally easily performed is performed thereon without the necessity of an atmosphere control and by heating thereof in the normal oxygen-containing atmosphere, with almost no change of the coated layer to be protected which mainly influences optical characteristics thereof.

According to a first aspect of the present invention, there is provided a method of making a heat treated coated glass comprising the steps of:

30 forming a solar control layer or an electroconductive layer on a glass substrate;  
 forming a first protective layer composed of a non-oxide or not completely oxidized, which is transparent in a region of visible light, and which remains transparent even when oxidized,  
 to provide a glass coated with a multi-layer comprising at least two layers including said solar control layer or an electroconductive layer and said first protective layer; and  
 35 performing heat-treatment for said coated glass.

According to a second aspect of the present invention, there is provided the method of making a heat treated coated glass according to the first aspect, wherein a second protective layer which is composed of a metal oxide and is transparent, is formed on the first protective layer, to provide a glass coated with a multi-layer composed of at least three layers including the solar control layer or an electroconductive layer, the first protective layer and said second protective layer, and then a heat-treatment is performed for said coated glass.

In the drawings:

Figure 1 is a sectional diagram of an example of heat treated coated glass which is made by the present invention, Figures 2 and 3, sectional diagrams showing other examples of heat treated coated glass according to the present invention.

45 In these Figures, a reference numeral 1 designates a solar control layer, 2, a first protective layer, 3, a second protective layer and 4, a glass substrate.

In the present invention, as the solar control layer 1, a film provided with a solar energy absorbing function having a light absorption characteristic at least with respect to a portion of a region of wave length of the solar ray, or a film provided with a light reflection characteristic mainly with respect to a region of near infrared ray, or the like can be used.

50 Specifically, a film whose major component is at least one of stainless steel, titanium, chromium, zirconium, tantalum and hafnium (hereinafter, defined as metal M), a nitride of the metal M, a boride of the metal M, a carbide of the metal M or a mixture of these, or a film whose major component is a metal such as silver, aluminium, chromium or the like, is pointed out. In case of the nitride, the boride, the carbide or the like, a thickness thereof is preferably approximately from 100Å to 800Å to obtain the solar control property. In case of the metal film, the film thickness is preferably 20Å to 300Å.

This invention is applicable to an electroconductive layer whose carriers are caused from the oxygen vacancies, in place of the solar control layer. As the examples of materials of such films, an indium oxide doped with tin, a tin oxide doped with antimony or fluorine, or a zinc oxide doped with aluminum, boron or silicon, etc., can be pointed out. Moreover,  $ZnO$ ,  $TiO_2$ ,  $SnO_2$ ,  $ThO_2$ ,  $V_2O_5$ ,  $Nb_2O_5$ ,  $Ta_2O_5$ ,  $MoO_3$ ,  $WO_3$ , 5  $MnO_2$ ,  $PbCrO_4$ , are also the examples of the materials of the above electroconductive layer. Hereinafter "solar control layer" represents not only the above solar control layer but also the above electroconductive layer in this specification.

The first protective layer 2 is a film which is a film of a non-oxide or a film that is not completely oxidized, and which is transparent in the visible light region and which is transparent in the visible light 10 region even when it is oxidized. The first protective layer 2 is a film which prevents diffusion of oxygen into the solar control layer 1 even when it is heated in the heat treatment, or, which does not discharge oxygen to the solar control layer 1 even when the first protective layer 2 per se contains oxygen. Since in the first protective film, only the refractive index thereof changes, the change of absorption is not caused and a transparency thereof is maintained even when it is oxidized, no considerable influence is applied on the 15 optical characteristics thereof after the heat treatment.

The most preferable example of the first protective layer 2, is a film whose major component is at least one of a boron nitride, a carbon nitride and a silicon nitride. Especially, a representative example is a film whose major component is a nitride of silicon or boron, or a film whose major component is a nitride of at 20 least two of silicon, boron, aluminium, zirconium and tin, particularly a film of a zirconium siliconnitride, a film of tin siliconnitride or the like. Or, it may be a film whose major component is a partially oxidized substance of the nitride, the boronnitride, the carbonnitride, the siliconnitride and the like. Among these, the silicon nitride and the zirconium siliconnitride are particularly provided with an excellent oxygen barrier performance. In the film of the nitride or the like as the above-mentioned first protective film, the invention is applicable thereto so far as it is transparent in the visible light region, even when the nitride is not bonded in the most general 25 stoichiometrical ratio. For instance, in case of the silicon nitride,  $Si_3N_4$  is the general composition. However, this invention is applicable so far as the film is transparent in the visible light region. The atomic ratio of N to Si is preferably 1.25 or more, because the silicon nitride film is transparent in the visible light region.

When the second protective layer 3 is formed as an outermost layer, the thickness of the first protective layer 2 depends on the oxygen barrier performance, and is determined in consideration of a quantity of 30 oxygen which is defused inside through the second protective layer. In the normal heat treatment, the thickness is preferably at least about  $10\text{\AA}$ , particularly  $20\text{\AA}$  or more.

In this invention, the effect of the invention can be provided even when the second protective film is not formed and the first protective film is formed with a thickness of the film which is thick to some degree. The film thickness should be determined in consideration of a film thickness of a portion of the film of which is 35 oxidized in the heat treatment.

The second protective layer 3 is preferably composed of a metal oxide, is a transparent film in the visible light region, and provided with a high oxygen barrier performance. Specifically, an oxide such as a tin oxide, a zinc oxide, a zirconium oxide, a tantalum oxide, a niobium oxide, a titanium oxide, a silicon oxide or the like is pointed out as the metal oxide. The thickness of the second protective layer 3 is 40 determined in consideration of the thickness of the first protective layer 2, and the optical characteristics in view of a total of the coated glass and the like.

In the present method, it is preferable to form a film which displays the same function with that in the first protective film, between the solar control film 1 and the glass substrate 4, to shield oxygen diffused from the glass substrate in a very small quantity, that is, a non-oxide film or a film that is not completely 45 oxidized, which is transparent even when it is oxidized, as a first underlayer 5, as shown in Figure 2.

As the materials of such first underlayer 5, the materials described for the first protective layer 2 can be similarly used.

In this invention, the solar control layer 1 to be protected can be perfectly protected from the oxidation during the heating process for bending and/or tempering, by the first protective layer 2, and, in some cases, 50 by the first protective layer 2 together with the first underlayer 5.

Furthermore, in the invented method, a film may be formed as a second underlayer 6 which is composed of a metal oxide and is transparent, between the glass substrate 4 and the first underlayer 5, as shown in Figure 3, to obtain a desired optical characteristic (transmittance, reflectance, color tone etc.) by employing interference of light, or to promote the durability such as the adherence. As the second 55 underlayer 6, the film exemplified as the first protective layer can be utilized. In this invention, the change of characteristics of the coated layers can be minimized, because the two metal oxide layers of the second protective layer 3 and the second underlayer 6 which have no change during the heating decrease the oxygen diffusing to the first protective layer 2 and first underlayer 5 which absorb said oxygen and change

only their refractive indices.

Furthermore, without being restricted by these films, a desired film may be formed between the glass substrate 4 and the first underlayer 5.

The glass substrate 4 is not particularly restrained. Various glass plates such as a soda-lime glass plate or a heat ray absorbing glass plate can be employed.

The bending method as the heat treatment is not particularly restrained and may be a normally performed method wherein coated glass is heated at approximately 580 to 700°C (which temperature depends on the composition of a glass) in the atmosphere and is bent. Naturally, it may be bent in a nonoxidizing atmosphere. However, in that case revamping of a device or the like is necessary and the production cost is increased. The tempering method as the heat treatment is also not particularly restrained and may be a normal method wherein coated glass is heated at approximately 500 to 700°C in the atmosphere and rapidly cooled. It is possible to perform the tempering continuously successive to the bending. The tempering may be a full-tempering or a semi-tempering.

In the heat treated coated glass, there may be caused almost no change in the composition of the first protective layer, since the oxidation thereof is not promoted in case wherein the oxygen barrier performance of the second protective film is excellent. However, the first protective layer often contains oxygen more than that before the heat treatment, since the oxidation thereof is promoted by the heating in the heat treatment. In some cases, it is converted into an oxide by discharging nitrogen or carbon in the first protective layer in the heat treatment. Or, when the second protective layer is not formed, some degree of oxidation is promoted from the surface of the first protective layer but the first protective layer is in a state wherein the oxidation thereof is not performed to a portion adjacent to the solar control layer.

The solar control layer 1 which covers glass, is normally oxidized in the heat treatment by oxygen in an atmospheric gas (the atmosphere) which is diffused to the inside thereof through the oxide film protecting the solar control layer 1 and by a combined oxygen in the contiguous oxide film, thereby considerably changing the optical characteristics thereof, particularly the heat ray shielding function, the visible light transmittance and the like (the electroconductivity in case of an electroconductive layer).

In this invention, as the second protective layer 3 for the protection, one which is provided with an excellent oxygen barrier performance is selected thereby considerably reducing the oxygen diffusing inside thereof. Furthermore, the first protective layer 2 is provided between the second protective layer 3 and the solar control layer 1. Accordingly, the first protective layer 2 mainly reacts with the oxygen diffused inwardly through the second protective layer in a very small amount or the combined oxygen in the second protective layer, thereby preventing the diffused oxygen and the combined oxygen to reach the solar control layer 1 to be protected. Therefore, the solar control layer 1 suffers almost no change.

Furthermore, even when the second protective layer 3 is not formed, the first protective layer sufficiently absorbs the oxygen from the atmosphere during the heat treatment and plays a role of an oxygen barrier. Therefore, the solar control layer 1 suffers almost no change.

Since the solar control layer 1 suffers almost no change in view of a total of the multi-layer coated on glass, although the refractive index of the first protective layer changes more or less, as a total of the film, there causes no change in the optical characteristics which takes place in the conventional heat treatment.

#### EXAMPLE 1

A reactive sputtering is performed on the plane glass substrate 4 employing a tin target in a mixture of oxygen and argon gases, thereby forming the second underlayer 6 (250Å) composed of a tin oxide. Next, another reactive sputtering is performed thereon employing a zirconium silicide alloy target in nitrogen gas, thereby forming the first underlayer 5 (50Å) composed of a zirconium siliconitride.

Next, a third reactive sputtering is performed thereon employing a chromium target in nitrogen gas thereby forming the solar control layer 1 (100Å) composed of a chromium nitride. Thereafter, the first protective film 2 (50Å) composed of a zirconium siliconitride similar to the first underlayer 5, and the second protective film 3 (300Å) composed of a tin oxide similar to the second protective layer 6 are formed. In this way, a coated glass having the construction as shown in Figure 3, is provided.

The plane coated glass is put into an electric furnace wherein it is heated at about 630°C in the atmospheric environment and the bending thereof is performed. There is no change in the solar control layer 1, the second protective layer 3 and the second underlayer 6 after the bending as compared with those before the bending. The first protective film 2 and the first underlayer 5 after the bending are partially oxidized. The optical characteristics of the coated glass before and after the heating are shown in Table 1.

## COMPARATIVE EXAMPLE 1

5 A tin oxide layer (250Å), a chromium nitride layer (100Å) and a tin oxide layer (300Å) are successively formed on a plane glass substrate by the sputtering method similar to Example 1. The heat treatment similar to that in Example 1 is performed on the obtained plane coated glass and the optical characteristics before and after the heating are measured. The result is shown in Table 1.

## COMPARATIVE EXAMPLE 2

10 10 A chromium nitride layer (100Å) is formed on a plane glass substrate by the sputtering method similar to that in Example 1. The heat treatment similar to that in Example 1 is performed on the obtained plane coated glass. The optical characteristics before and after the heating thereof are shown in Table 1.

Table 1

		Color coordinate according to CIE 1931 (XYZ)			Color difference before & after heating according to CIE 1976 (L* <sup>a</sup> <sub>b</sub> <sup>v</sup> )	
		Visual light transmittance: Y	x	y		
15						
20						
25	Coated glass of Example 1	Before bending After bending	44.7% 46.0%	0.299 0.297	0.307 0.306	- 1.67
30	Coated glass of Comparative Example 1	Before bending After bending	46.9% 51.8%	0.304 0.308	0.309 0.312	- 3.97
	Coated glass of Comparative Example 2	Before bending After bending	30.4% 78.6%	0.317 0.314	0.323 0.321	- 29.19

## EXAMPLE 2

35 A coated glass as in Figure 3 (that is, a coated glass having a construction of glass substrate/tin oxide layer (320Å)/silicon nitride layer/chromium nitride layer (136Å)/silicon nitride layer/tin oxide layer (280Å)), is obtained by a method similar to Example 1 except that the silicon nitride layers are formed as the first underlayer 5 and the first protective layer 2, by performing a reactive sputtering employing silicon targets in 40 nitrogen gas.

45 The changes of the optical characteristics are shown in Table 2, in cases wherein the heat treating temperature is varied with respect to three kinds of film thicknesses, namely 30Å, 20Å and 0Å (that is, a case wherein the silicon nitride is not formed) of the silicon nitride layers as the first underlayer 5 and the first protective layer 2. The atomic ratio of nitrogen to silicon in the silicon nitride layers in Example 2 was 1.35.

50 By the comparison of the results of Example 1, Comparative Examples 1 and 2, and Table 2, it is found that the coated glass of this invention suffers little change with respect to the optical characteristics thereof in the heat treatment in the atmosphere and the significance of the invented method is clarified.

Table 2

5 10 15 20 25 30	Film thickness of silicon nitride (Å)	Heating temperature (°C)	Color coordinate according to CIE 1931 (XYZ)			Color difference before & after heating according to CIE 1976 (L*U*V*)
			Visible light transmittance: Y (%)	x	y	
30	30	Before heating	48.57	0.2973	0.3207	-
		530	48.39	0.2975	0.3186	1.46
		600	49.15	0.2976	0.3186	1.51
		620	49.91	0.2987	0.3201	1.45
		640	49.73	0.2981	0.3189	1.69
		660	48.80	0.2987	0.3193	1.59
20	20	Before heating	51.44	0.2999	0.3225	-
		530	51.13	0.2992	0.3198	1.67
		600	51.04	0.3010	0.3210	1.74
		620	53.85	0.3007	0.3209	1.93
		640	53.69	0.3008	0.3207	2.02
		660	51.56	0.3035	0.3226	2.50
0	0	Before heating	46.67	0.2962	0.3170	-
		530	49.58	0.2996	0.3185	2.70
		600	51.04	0.3001	0.3185	3.50
		620	52.01	0.3000	0.3192	3.92
		640	53.20	0.3002	0.3192	4.57
		660	54.58	0.3003	0.3190	5.27

This invention has an advantage wherein the coating can be performed by the normally utilized coating device for a plane glass substrate. Therefore, the coating control is extremely easy, whereby a glass having the high quality and covered with a uniform film can be obtained at a low cost.

Furthermore, in this invention, since the heat treatment can be performed in the atmosphere dispensing with an atmospheric control, the fabrication cost is extremely inexpensive and there is no change in the optical characteristics before and after the heat treatment. Therefore, in case of employing a plane glass and a bent glass and/or a tempered glass in combination thereof, the same kind of coated glass, that is, a non-heat-treated one for the former and a bent and/or tempered one for the latter, can be employed. Accordingly, it is not necessary to prepare a plane glass having optical characteristics after they are changed by the heat treatment, with a film having a construction different from those in a bent glass or a tempered glass, as in the conventional case.

## Claims

1. A method of making a heat treated coated glass comprising the steps of:
  - forming a solar control layer or an electroconductive layer on a glass substrate;
  - forming a first protective layer composed of a non-oxide or not completely oxidized, which is transparent in a region of visible light, and which remains transparent even when oxidized,
  - to provide a glass coated with a multi-layer comprising at least two layers including said solar control layer or an electroconductive layer and said first protective layer; and
  - performing heat-treatment for said coated glass.
2. The method of making a heat treated coated glass according to Claim 1, wherein a second protective layer which is composed of a metal oxide and is transparent, is formed on the first protective layer, to provide a glass coated with a multi-layer composed of at least three layers including the solar control layer or an electroconductive layer, the first protective layer and said second protective layer, and then a heat-treatment is performed for said coated glass.

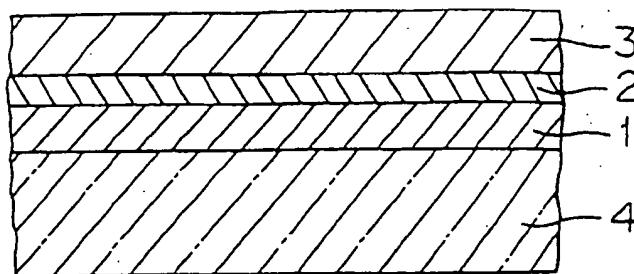
3. The method of making a heat treated coated glass according to Claim 1, wherein the solar control layer is a film whose major component is at least one of a metal (hereinafter, defined as metal M) selected from the group consisted of stainless steel, titanium, chromium, zirconium, tantalum and hafnium, or a nitride of said metal M, or a boride of the metal M, or a carbide of the metal M or a mixture of these.
- 5
4. The method of making a heat treated coated glass according to Claim 1, wherein the first protective layer is a film whose major component is at least one selected from the group consisted of a nitride, a boron nitride, a carbon nitride and a silicon nitride.
- 10
5. The method of making a heat treated coated glass according to Claim 1, wherein the first protective layer is a film whose major component is a nitride of silicon or boron, or a nitride of at least two selected from the group consisted of silicon, boron, aluminum, zirconium and tin.
- 15
6. The method of making a heat treated coated glass according to Claim 2, wherein the second protective layer is a film having an oxygen barrier performance.
7. The method of making a heat treated coated glass according to Claim 2, wherein the second protective layer is a film whose major component is at least one selected from the group consisted of a tin oxide, a tantalum oxide, a titanium oxide, a niobium oxide, a titanium oxide and a silicon oxide.
- 20
8. The method of making a heat treated coated glass according to Claim 1, wherein a first underlayer composed of a non-oxide or not completely oxidized, and which remains transparent even when oxidized, is formed on the glass substrate before said solar control layer is formed on the glass substrate.
- 25
9. The method of making a heat treated coated glass according to Claim 8, wherein a second underlayer which is composed of a metal oxide and is transparent, is formed on the glass substrate before said first underlayer is formed on the glass substrate.
- 30
10. The method of making a heat treated coated glass according to Claim 1, wherein said heat-treatment is a process of heating before bending and/or tempering.
11. A coated glass comprising:
  - a glass substrate having a curved configuration or a tempered glass substrate, and
  - 35
  - a multi-layer coating comprising at least two layers of an unoxidized solar control layer or an electroconductive layer and a first protective layer which is transparent in a region of visible light and composed of a non-oxide or an oxide, coated on said glass substrate.
12. The coated glass according to Claim 11, wherein a second protective layer which is composed of a metal oxide and is transparent, is formed on the first protective film.
- 40
13. The coated glass according to Claim 11, wherein the first protective layer is a film whose major component is at least one selected from the group consisted of a

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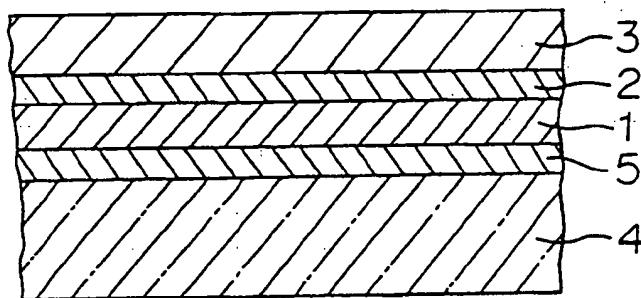
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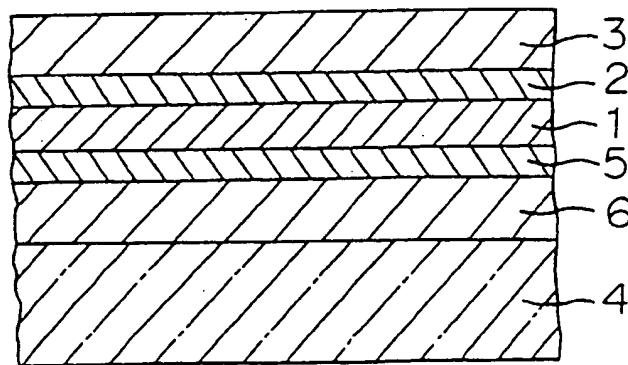
**FIGURE 1**



**FIGURE 2**



**FIGURE 3**





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PAGE1

DOCUMENTS CONSIDERED TO BE RELEVANT		Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl.5)
Category	Citation of document with indication, where appropriate, of relevant passages		
X	PATENT ABSTRACTS OF JAPAN vol. 13, no. 49 (C-565)3 February 1989 & JP-A-63 242 948 ( ASAHI GLASS CO LTD ) 7 October 1988 * abstract * ---	1-10	C03C17/34 C03C17/36
X	PATENT ABSTRACTS OF JAPAN vol. 14, no. 544 (C-784)4 December 1990 & JP-A-02 233 534 ( CENTRAL GLASS CO LTD ) 17 September 1990 * abstract *---	1-10	
X	DE-A-3 942 990 (LEYBOLD AG) * claims 1-5; figure 1 *---	1-2,5-7, 9	
X	DE-A-3 941 797 (LEYBOLD AG) * claims 1-7; figure 1 *---	1-2,5-7, 9,11-13	
X	EP-A-0 402 075 (NIPPON SHEET GLASS CO) * claims; figure 1A *---	1-2,5-7, 9	TECHNICAL FIELDS SEARCHED (Int. Cl.5)
X	PATENT ABSTRACTS OF JAPAN vol. 12, no. 496 (C-555)23 December 1988 & JP-A-63 206 333 ( ASAHI GLASS CO LTD ) 25 August 1988 * abstract *---	1-10	C03C
X	PATENT ABSTRACTS OF JAPAN vol. 13, no. 402 (C-633)6 September 1989 & JP-A-01 145 351 ( ASAHI GLASS CO LTD ) 7 June 1989 * abstract *---	1-2,5-7, 9	
The present search report has been drawn up for all claims		Examiner	KUEHNE H.C.
Place of search	Date of completion of the search		
BERLIN	26 FEBRUARY 1993		
CATEGORY OF CITED DOCUMENTS			
X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document			
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PAGE2

DOCUMENTS CONSIDERED TO BE RELEVANT									
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A	PATENT ABSTRACTS OF JAPAN vol. 14, no. 427 (C-758)13 September 1990 & JP-A-02 164 744 ( ASAHI GLASS CO LTD ) 25 June 1990 * abstract * ---	1-2, 5-7, 9							
A	PATENT ABSTRACTS OF JAPAN vol. 13, no. 72 (C-570)17 February 1989 & JP-A-63 265 844 ( NIPPON SHEET GLASS CO LTD ) 2 November 1988 * abstract * ---	1-13							
A	PATENT ABSTRACTS OF JAPAN vol. 14, no. 522 (C-778)15 November 1990 & JP-A-02 217 339 (ASAHI GLASS CO LTD ) 30 August 1990 * abstract * ---	1-10							
			TECHNICAL FIELDS SEARCHED (Int. Cl.5)						
<p>The present search report has been drawn up for all claims</p> <table border="1"> <tr> <td>Place of search</td> <td>Date of completion of the search</td> <td>Examiner</td> </tr> <tr> <td>BERLIN</td> <td>26 FEBRUARY 1993</td> <td>KUEHNE H.C.</td> </tr> </table> <p>CATEGORY OF CITED DOCUMENTS</p> <p>X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document</p> <p>T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons ..... &amp; : member of the same patent family, corresponding document</p>				Place of search	Date of completion of the search	Examiner	BERLIN	26 FEBRUARY 1993	KUEHNE H.C.
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